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DEVELOPMENTAL EVALUATION OF NEAR 17°K MOLECULAR SIEVE 5A FOR HYDROGEN CRYOSORPTION

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ARO, Inc.

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AEROSPACE ENVIRONMENTAL FACILITY
ARNOLD ENGINEERING DEVELOPMENT CENTER
AIR FORCE SYSTEMS COMMAND
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NOTICES

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MOLECULAR SIEVE 5A FOR HYDROGEN CRYOSORPTION

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FOREWORD

The results of research reported herein were obtained by ARO, Inc. (a subsidiary of Sverdrup and Parcel, Inc.), contract operator of the Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC), Arnold Air Force Station, Tennessee, under Contract AF 40(600)-1000, Program Element 65402234/7778, Task 777801. The research was conducted from February to June, 1964 under ARO Project No. SN2304, and the report was submitted by the author on February 11, 1965.

This technical report has been reviewed and is approved.

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ABSTRACT

Test results are presented for the cryosorption of hydrogen on a commercially manufactured molecular sieve 5A panel operated near 20°K. The effects of repeated activations, water and air contamination under vacuum, and exposure to atmospheric conditions were observed. Using suitable operating techniques, pumping speeds near theoretical maximums were obtained.

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ILLUSTRATIONS

SECTION I
INTRODUCTION

Clean vacuum production is a significant requirement for space simulation facilities. Cryopumps operating near 20°K satisfy cleanliness requirements and provide high volumetric pumping speeds for all gases and vapors except hydrogen, helium, and neon. The current practice of diffusion pumping these noncondensables can impose undesirable environmental contaminants, particularly pump oil. Pumping speeds limited by available chamber wall area and economic factors further minimize advantages of diffusion pumping for large installations.

Several methods for hydrogen and helium gas removal are currently under investigation. Low temperature physical adsorption (cryosorption), and various forms of gettering are being evaluated. Since cryopumping techniques are used to pump the majority of gases, the incorporation of cryosorption pumping at a small additional cost is attractive.

Recent research (Refs. 1 and 2) indicates molecular sieve 5A, a silicate base zeolite, exhibited desirable cryosorption properties. A sample of this material was evaluated to determine its suitability for use in space chambers. The desired information included pumping speed and capacity of the material, effects of common space chamber contaminants on pumping speed, and mechanical integrity of the material in a typical space chamber installation.

SECTION II
APPARATUS, INSTRUMENTATION, AND INSTALLATION

2.1 TEST CHAMBER

Evaluation of the molecular sieve 5A was conducted in the Aerospace Research Chamber (7V), Fig. 1. This chamber is 7 ft in diameter and 12 ft in length. A liquid-nitrogen-cooled chamber liner (6 ft in diam by 12 ft long) is refrigerated by a 50-kw pressurized nitrogen system. A 1-kw gaseous helium refrigeration system provides coolant to cryopanels as required for tests, and was used to cool the molecular sieve 5A sample in the range of 13 to 30°K. Two 32-in. oil diffusion pumps backed by two 6-in. pumps provided additional pumping capacity.

2.2 TEST SURFACE

A 300-g adsorbent sample was used in the study, Fig. 2. This sample was bonded to the smooth side of a 29- by 47-in. single embossed stainless steel panel. The panel was edged with a stainless steel strip which limited the exposed sieve area to 8.35 ft^2. This assembly, with a 1/16-in.-thick adsorbent tightly bonded to the surface, provided a panel through which gaseous helium could be circulated. (A 0.019-in. 16 by 16 mesh stainless steel screen intermittently brazed to the panel was imbedded in the sieve material to provide additional strength and thermal cooling. Portions of this screen were visible in the sieve, resulting in a small area reduction in the pumping surface.)

A means of liberating preadsorbed gases from the adsorbent is required. Resistive heating, provided by six 500-w strip heaters mounted on the embossed side of the panel as shown in Fig. 2, was used for this purpose.

2.3 TEST INSTRUMENTATION

The apparatus used to introduce hydrogen and helium test gas into the chamber is shown in Fig. 3. Two constant conductance leaks made from porous sintered stainless steel plugs metered the gases. Reference 3 describes the leaks and their calibration process. Periodic calibration throughout the tests confirmed constant conductance values of the leaks.

Chamber pressures were determined by nude ionization gages and a mass spectrometer. In the initial test phase, the ionization gages were calibrated using a known orifice calibration system (Ref. 4), and mass spectrometer sensitivities were determined by comparison with the calibrated gages. In succeeding tests in-chamber gage calibrations were made by a pressure rate-of-rise method using the known chamber volume and calibrated leaks.

Six copper-constantan thermocouples attached to the panel were used to monitor activation temperatures. Cryogenic operating temperatures were determined from hydrogen vapor pressure thermometers located in the panel inlet and outlet lines.

2.4 TEST INSTALLATIONS

The test program was accomplished in two test chamber entries. These test periods were separated by 2-1/2 months during which time the panel was stored in an unconditioned area.

2.4.1 Phase I

Phase I testing was conducted in conjunction with a molecular kinetics study (Ref. 5). The chamber volume in which the panel was installed was reduced by the installation of a liquid-nitrogen-cooled wall in the chamber so that the test volume was approximately 6 ft in diameter and 7 ft long. The cryosorption panel without the molecular kinetics model in place is shown in Fig. 4. Figure 5 shows both test installations in place and the associated instrumentation.

2.4.2 Phase II

In Phase II, the adsorbent panel was installed as shown in Fig. 6. The entire chamber was used. During this test, the diffusion pumps were not exposed directly to the test volume unless the 2-ft-diam separating valve was opened. The valve was opened during panel activation to permit the 32-in. pumps to remove the 77°K noncondensable gases desorbed during this period. A copper cryopanel, cooled by exhaust helium from the cryo-sorption panel, extended the length of the test section. All openings in the LN_2-cooled liner were sealed with aluminum foil. Thus, the cryosorption panel was surrounded by a clean test volume pumped by the adsorbent, a 20°K cryosurface, and the 77°K wall. Gas was admitted into the chamber through the diffusing ball shown in Fig. 6.

SECTION III
PROCEDURE

3.1 DEFINITION OF PARAMETERS

The equation describing the volumetric pumping speed reduces from the Perfect Gas law and the conservation of mass to

$$S = \frac{Q}{P_p - P_B}$$

where

S = volumetric pumping speed (ℓ/sec)

Q = test gas flow rate (torr ℓ/sec)

P_p = Pumping pressure with flow rate imposed (torr)

P_B = Residual pressure attainable after the test gas flow was terminated. In all cases this was negligible.

Two types of pressure-time curves were observed, depending on the gas flow rate. For small flows, typical constant pressure pumping was observed as shown below.
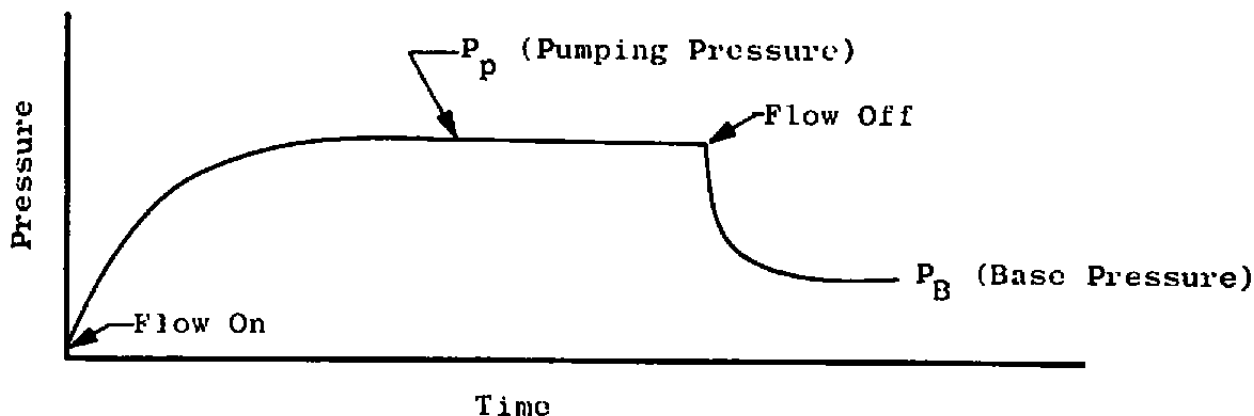
As shown in the next sketch, higher flow rates were accompanied by continually increasing pumping pressures as the adsorbent loading increased.
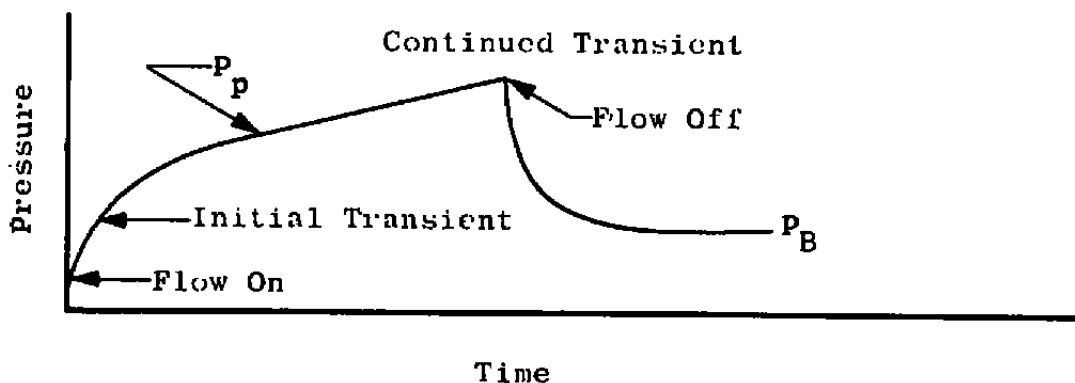
The pumping pressure used to compute the pumping speed for the transient case was the point at which the slope of the pressure-time curve became constant after the test gas flow was started. This occurred immediately after the initial transient subsided. In all cases reported herein less than five percent of the test gas flow is represented by the pressure-time transient; that is, 95 percent of the metered flow was being pumped as the measurements were made.

3.2 GENERAL PROCEDURE

The general test procedure used in both phases was as follows:

1. The chamber was rough pumped using the mechanical pump.

2. Diffusion pumps were started and all LN$_2$ surfaces were cooled.

3. The adsorbent material was activated by heating. A typical activation cycle is shown in Fig. 7.

4. After activation, the cryosorption panel was cooled to operating temperature by a flow of gaseous helium.

5. Test gas was introduced into the chamber at various flow rates. By measuring the resulting chamber pressures at each flow rate, the pumping speed of the adsorbent could be computed. A test run was constituted by varying the flow rates over a wide range for each adsorbent temperature case.

3.3 TEST SEQUENCE

3.3.1 Phase I

The test sequence of Phase I was as follows:

1. Pumping speed of the 32-in. oil diffusion pumps was determined before panel activation.

2. The panel was activated and hydrogen pumping speeds were determined with both the 32-in. pumps and panel operating.

3. Four thousand atm cc of water vapor were deposited on the panel and hydrogen pumping speed measurements repeated. No activation was conducted between steps 2 and 3.

3.3.2 Phase II

The following test sequence was used in Phase II:

1. The minimum pressure attainable in the test volume was determined following the adsorbent panel activation and cooldown of all cryosurfaces.

2. Hydrogen pumping speed data (Run 1) were obtained with the panel at 17.5°K.

3. Vacuum conditions from Run 1 were maintained and the sieve was reactivated. Hydrogen pumping speed data were obtained with a 20.0°K panel (Run 2).

4. The panel was reactivated under continuous vacuum, cooled to 18.5°K, and hydrogen pumping speed data were obtained with various amounts of water vapor contaminant on the sieve (Run 3). The 77°K cell liner was not cooled during this run.

5. Panel reactivation and cooldown to 16.4°K preceded 77°K hydrogen pumping speed measurements with successive amounts of atmospheric air contaminants admitted to the panel (Run 4).

6. The panel was reactivated and the pumping speed for 77°K helium determined with a 13.6°K panel temperature (Run 5).

7. Following the helium run, and without a panel activation, 77°K hydrogen pumping speed with a 13.6°K panel was determined (Run 6).

8. The chamber was brought to atmospheric conditions and the sieve 5A exposed for approximately 36 hr. Cell pumpdown, adsorbent activation, and cooldown preceded measurement of hydrogen pumping speed with 17°K adsorbent (Run 7).

SECTION IV
RESULTS

4.1 PHASE I

Because of insufficient helium flow to the cryosorption panel, the desired operating temperatures were not obtained in Phase I. However, a hydrogen pumping speed of 4000 ℓ/sec for the panel was observed even with an average panel temperature of 34°K. Helium refrigerant flow was continued during the water vapor deposition period, resulting in a mean panel temperature decrease of 9°K before the next set of data was taken. Pumping speeds measured then were similar to values obtained before contamination (Fig. 8).

4.2 PHASE II

A sufficient helium refrigerant flow was provided such that the adsorbent panel inlet-outlet temperature differential was less than 1/2°K. One exception occurred. During and after the water vapor deposition, radiation heat loads from the ambient temperature surroundings caused a 1°K differential.

4.2.1 Runs 1, 2, and 7 — 77°K Hydrogen Adsorption on Molecular Sieve 5A

Three sets of 77°K hydrogen pumping speed data are presented in Fig. 9. The data represent three runs, 1, 2, and 7, with essentially two temperature conditions and the following activation histories.

Prior to Run 1, the adsorbent panel had been stored 2-1/2 months. The panel was installed and activated, and hydrogen pumping speeds were determined. At the completion of this test, vacuum conditions were maintained and the panel reactivated prior to conducting Run 2. Prior to Run 7, after Run 2, the chamber was opened and the adsorbent exposed to atmospheric conditions before activation.

Pumping speeds of 8.7 x 10⁴ ℓ/sec were observed during Run 1 in the low pressure ranges with sieve temperatures near 17°K. A correction for the constant speed of the 32-in. diffusion pumps is included in Run 1 data; in succeeding runs these pumps were not operated.

All runs exhibited an exponentially decreasing pumping speed with increasing flow rates. Some form of pumping speed decrease would be expected with increased panel loadings, but to demonstrate that the decrease in pumping speed was not entirely dependent on panel loading, the data in Run 2 were taken in the sequence shown in Fig. 9. A recovery in pumping speed is apparent after the reduction in hydrogen flow rate from Points 2 and 3. Constant pressure pumping was observed until the 20°K adsorbent was loaded with 33.4 atm cc of hydrogen.

As mentioned earlier, transient pumping accompanied high mass flow rates and large panel loadings. Figure 10, which is a continuation of Run 2 to large gas loadings, indicates the magnitude of this transient. Pumping speeds were calculated at various cumulative mass loadings while maintaining hydrogen flow rate at 0.72 torr ℓ/sec. The 6-in. diffusion pumps (200 ℓ/sec) were closed after a mass loading of 2.5 atm ℓ and the remaining data were obtained with only cryosorption pumping. Base pressure after admission of 8 atm ℓ of hydrogen was 1.3 x 10⁻⁵ torr measured 15 min after terminating the gas inflow. The same flow rate was again initiated and no pumping speed recovery was noted.

A performance degradation was observed in each succeeding run under continuous vacuum, apparently resulting from the effects of repeated activations. Pumping speed values determined in Run 7 indicated that adsorbent performance could be restored by exposing the material to atmospheric conditions prior to activation and use.

Isotherm data for Run 7 (Fig. 11) indicate the capacities provided in the high and ultrahigh vacuum regions.

4.2.2 Run 3 — 300°K Hydrogen Adsorption on Water-Contaminated Sieve 5A

The LN$_2$ surfaces were not cooled and the diffusion pumps were not operated during Run 3. This ensured that the water vapor contamination would deposit on the 20°K surfaces. Since the interior liner was not cooled, the radiation heat load from this source limited the cryosorption panel outlet temperature to 18.5°K.

A tare point (No. 1 in Fig. 12) was taken to establish the initial pumping speed with the adsorbent uncontaminated. A following point (No. 2) was taken with a combined hydrogen and water vapor inbleed which provided a total cell pressure in the low 10^{-5} torr range. Hydrogen partial pressure was determined by a mass spectrometer; at the time of measurement, 360 atm cc of water vapor had been admitted. Hydrogen flow was then terminated and the water flow was continued until 665 atm cc had been admitted. Two additional data points were determined which yielded pumping speed values in excess of the maximum theoretical rate of strike on the sieve area.

4.2.3 Run 4 — 77°K Hydrogen Pumping on Air-Contaminated Sieve 5A

Following panel activation and cooldown, three tare points were determined (Fig. 13). Then a 465-atm cc air sample containing an unknown amount of water vapor was admitted. A ten-fold decrease in hydrogen pumping speed was observed. Air inbleed was continued until 6850 atm cc had been admitted and an increase in pumping speed was observed.

4.2.4 Run 5 — 77°K Helium Adsorption on 13.6°K Molecular Sieve 5A

The feasibility of 77°K helium adsorption with sieve 5A was determined. As shown in Fig. 14, pumping speeds of 400 ℓ/sec were observed. Only cryogenic pumping was used during this run, and all pumping speed measurements were made at constant pressure. During the test, 14 atm cc of helium were adsorbed. Residual pressure attained after this amount of gas adsorbed was 1.5×10^{-7} mm Hg.

4.2.5 Run 6 — 77°K Hydrogen Adsorption with 13.6°K Molecular Sieve 5A

Hydrogen pumping speeds were determined with the adsorbent at 13.6°K with no activation following Run 5. Unexpectedly low values near 2.0×10^3 ℓ/sec were observed (Fig. 15), possibly resulting from the effects of preadsorbed helium. The capacity was significantly high; a residual hydrogen pressure of 1.8×10^{-7} torr was observed with 27,200 atm cc adsorbed.

Upon completion of the tests, the panel was examined for structural integrity and other observable changes. The material remained attached

to the substrate with no cracking or flaking. No other visual changes were noted in color or physical appearance.

SECTION V
DISCUSSION

5.1 HYDROGEN ADSORPTION ON MOLECULAR SIEVE 5A

Hydrogen pumping speeds on freshly activated sieve 5A at pressures of 10^{-8} torr were found to be about half the theoretical maximum rate of strike. This was demonstrated on two runs (Fig. 9) and represents hydrogen pumping speeds that compare quite favorably with diffusion pumps of comparable area. As hydrogen flow rates (i. e., rates of molecular strike) were increased, the high pumping speed advantage disappeared until, at a pressure of 10^{-5} torr, the pumping speed was only 5 percent of the theoretical maximum. Clearly, several other means of hydrogen pumping could be more attractive in these pressure ranges.

The trend of decreasing pumping speed with increasing rate of molecular strike indicates a limiting adsorption mechanism. Possibly this mechanism is diffusion. In any case, the pumping speed decrease could be minimized by using special extended surfaces, for example, deep corrugations or parallel panel arrays coated with adsorbent.

5.2 DEGRADATION OF PERFORMANCE WITH ACTIVATION

In general, a performance degradation was observed with repeated panel activations if vacuum conditions were maintained between successive runs. Data in Fig. 9 and tare points taken before Runs 3, 4, and 6 bear this out. However, it was found that the pumping speed could be recovered after an atmospheric exposure. Both phenomena have been observed in other work (Refs. 2 and 6), and an explanation has been presented based on catalytic action in the sieve material.

Although undesirable, these characteristics appear predictable. In Fig. 9, the two sets of pumping speed data preceded by an atmospheric exposure are nearly identical. References 2 and 6 also report that pumping speeds after atmospheric exposure and activation tend toward a common value. Furthermore, if a repeated activation is required to restore hydrogen cryosorption properties, it is reasonable to assume that such an activation as used in this study would be unnecessary. An activation at 100°K for a short time, depending on the available hydrogen pumping capability, should fully restore the adsorbent properties without an atmospheric exposure.

The initial activation schedule would be expected to affect the hydrogen pumping speed. References 1, 2, and 6 indicate that the primary contaminant after atmospheric exposure is water vapor. Gareis and Pitlor (Ref. 6) suggest that satisfactory cryosorption pumping with molecular sieve 5A can be obtained if the adsorbed water vapor is reduced to a maximum of 5 percent by weight. In the presence of a large water vapor pumping capacity, such as liquid-nitrogen-cooled panels, activation times could be reduced to the order of minutes at 400°F.

5.3 EFFECTS OF CONTAMINANTS ON HYDROGEN PUMPING SPEEDS

The experimental observations of this study seem to conflict with previous results regarding the effects of water vapor contamination. However, the method of application must be considered. Reference 2 reports that water vapor contamination was accomplished by exposure to atmospheric air. Various water loadings from 6.5 to 15.9 percent by weight were obtained. As the water contamination was applied as a frost in this study, it is thus indicated that hydrogen adsorption by water vapor depends on the method of water vapor application. This phenomenon was investigated further, and the results are reported in Ref. 7.

The significance of the nitrogen contamination data is not fully understood. Initially, after the admission of a small amount of air, pumping speed dropped as expected, but after a large amount of air was introduced the pumping speed increased. Possibly the combined adsorption effects of water vapor and condensed nitrogen could account for the recovery. This is supported by the conclusions of Hunt et al. (Ref. 8) and Hemstreet et al. (Ref. 9). However, further investigation would be required to support this conclusion.

<div align="center">

SECTION VI

CONCLUSIONS

</div>

The following conclusions are made on the basis of the experimental results:

1. Hydrogen pumping speeds near the theoretical maximum rate of strike can be realized on molecular sieve 5A.

2. Pumping speeds and capacities for hydrogen are related to operating temperatures and past history.

3. Helium can be successfully pumped with molecular sieve 5A.

4. Water vapor prevents cryosorption by molecular sieve 5A, but water vapor applied under vacuum on near 20°K surfaces has a significant adsorbent capacity for hydrogen.

5. Twenty-deg (K) condensables seriously degrade adsorbent performance.

6. Resistive heating is an acceptable and convenient activation method.

7. Mechanical integrity of the sieve-substrate combination is sufficient to withstand shipping, handling, thermal cycling, and repeated usage.

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9. Hemstreet, R. A., et al. "Research Study of the Cryotrapping of Helium and Hydrogen During 20°K Condensation of Gases, Phases I and II." AEDC-TDR-63-127, May 1963.

ULTIMATE PRESSURE - 1×10^{-10}

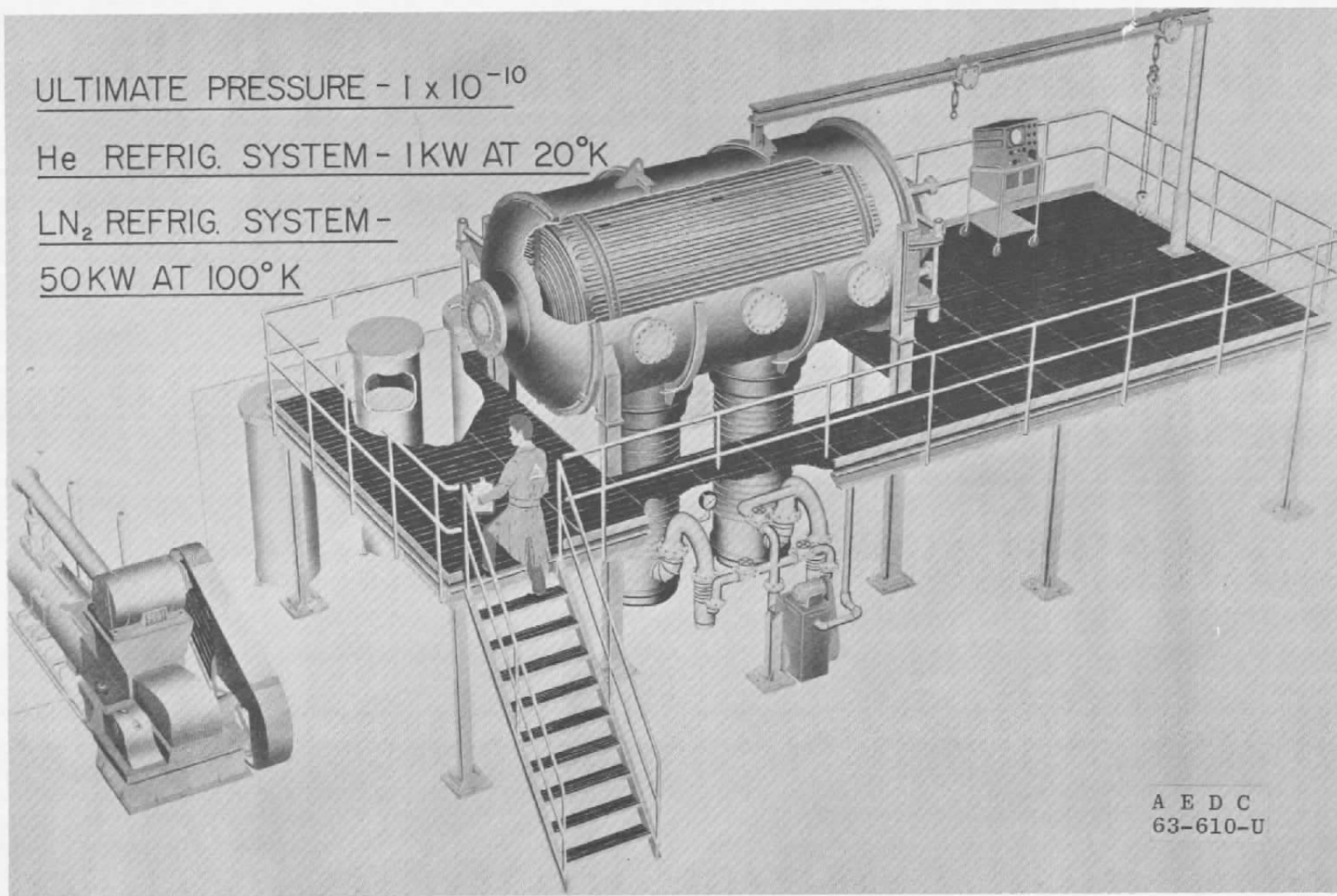He REFRIG. SYSTEM - 1KW AT $20°$K

LN$_2$ REFRIG. SYSTEM -

50 KW AT $100°$K

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63-610-U

Fig. 1 Aerospace Research Chamber (7V)

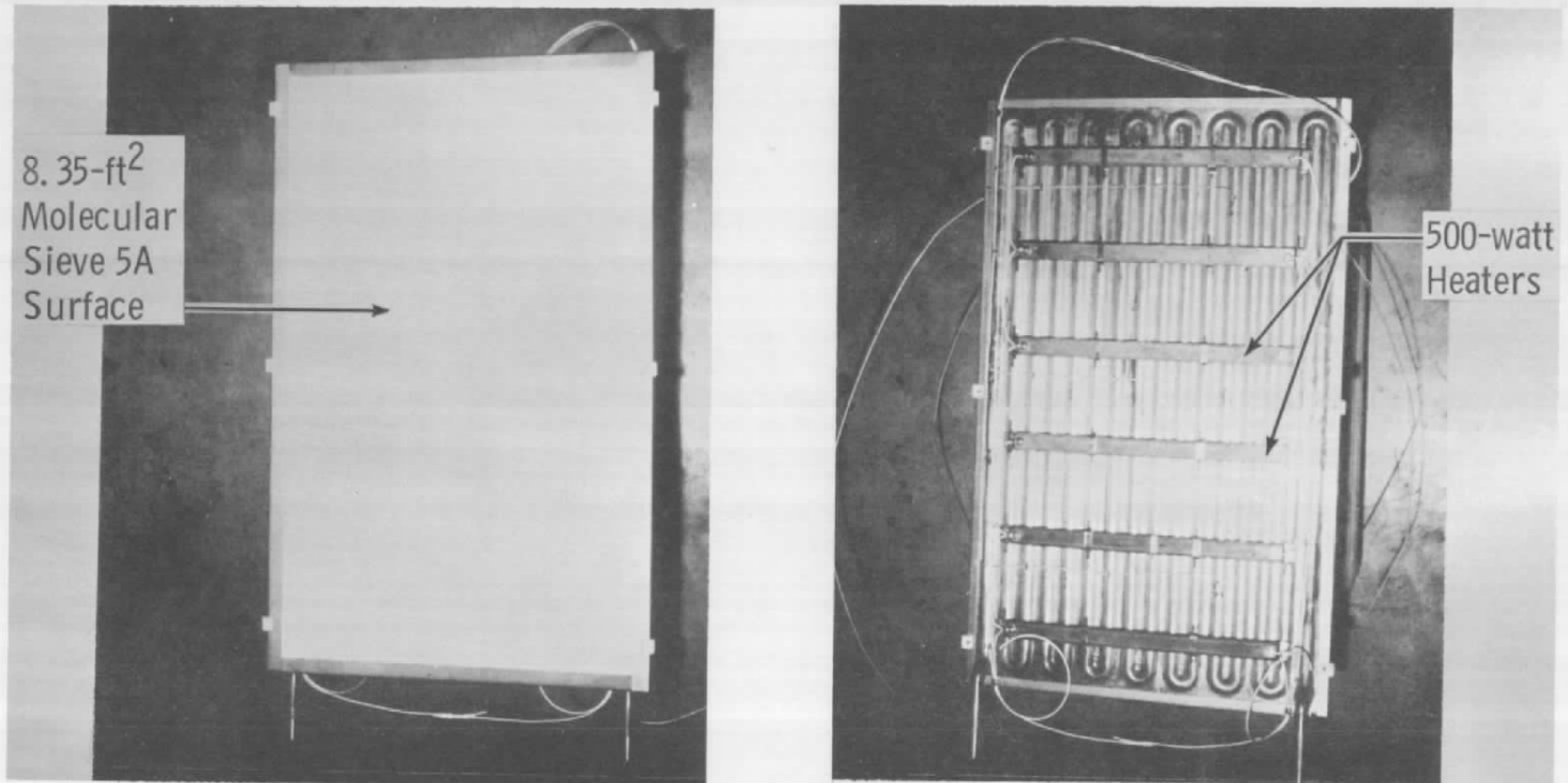8.35-ft^2 Molecular Sieve 5A Surface

500-watt Heaters

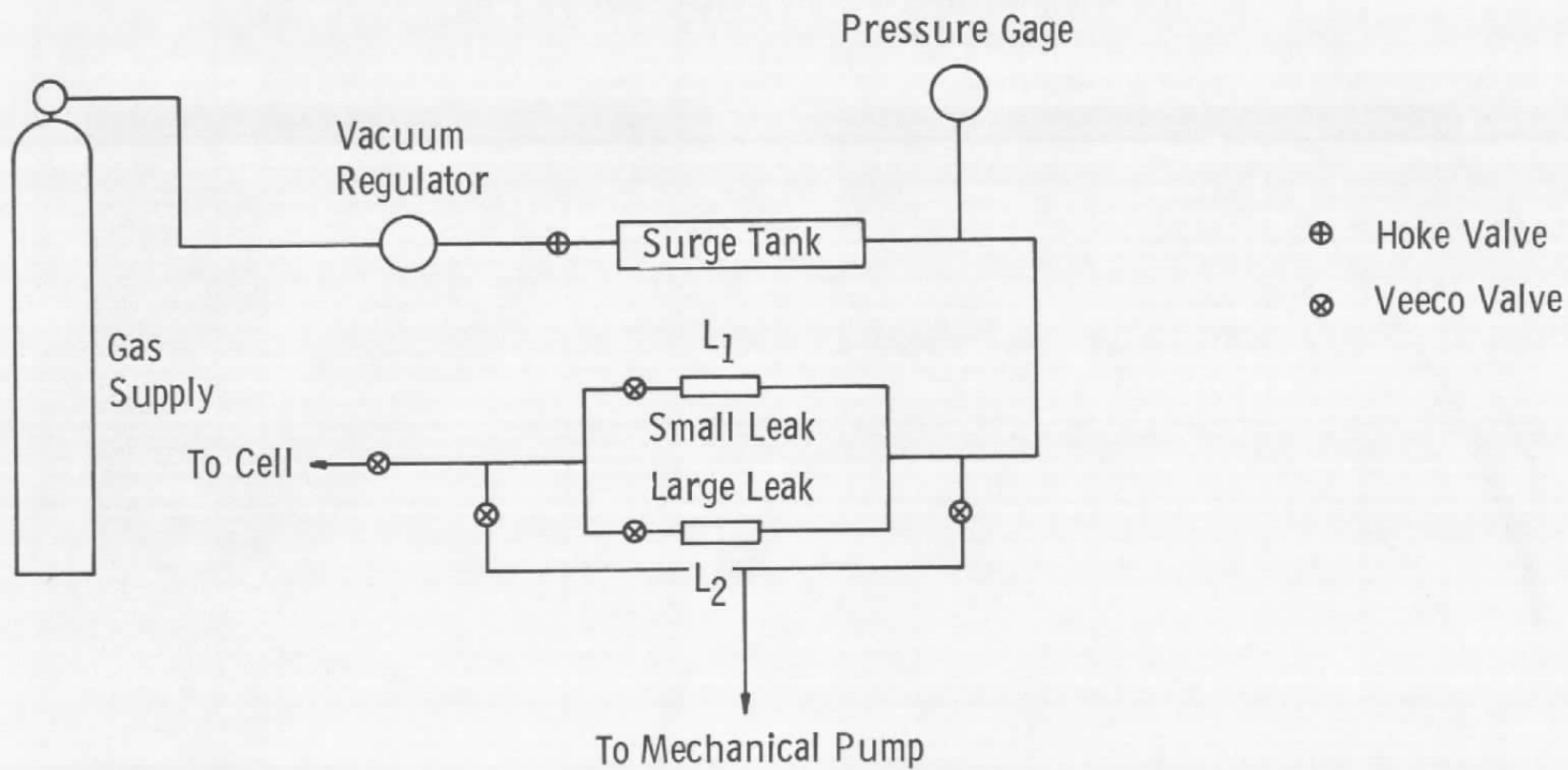Fig. 2 Adsorbent Panel and Activation Heater Installation
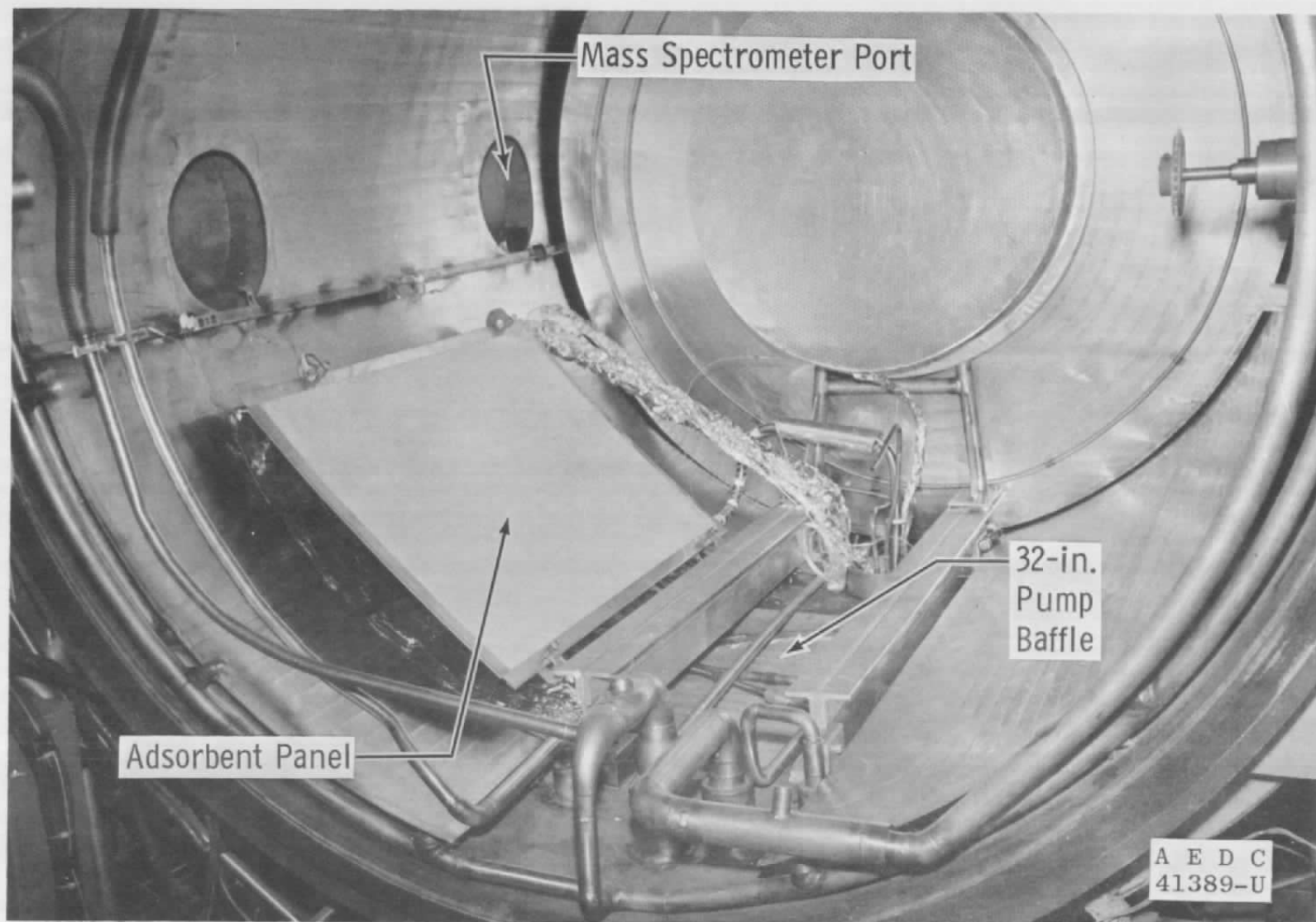
Fig. 3 Gas Inbleed System
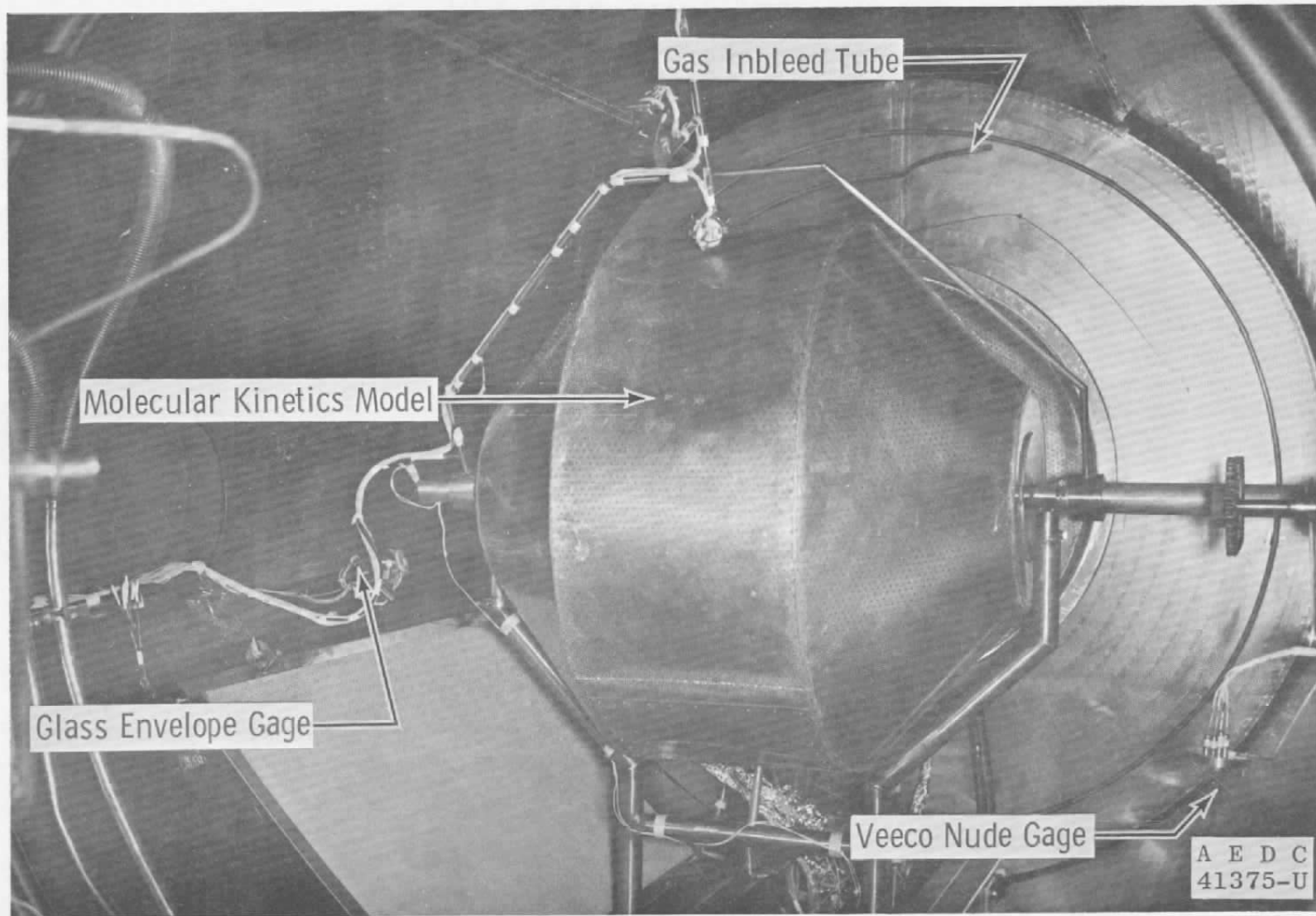
Fig. 4 Phase I Installation Less Molecular Kinetics Model
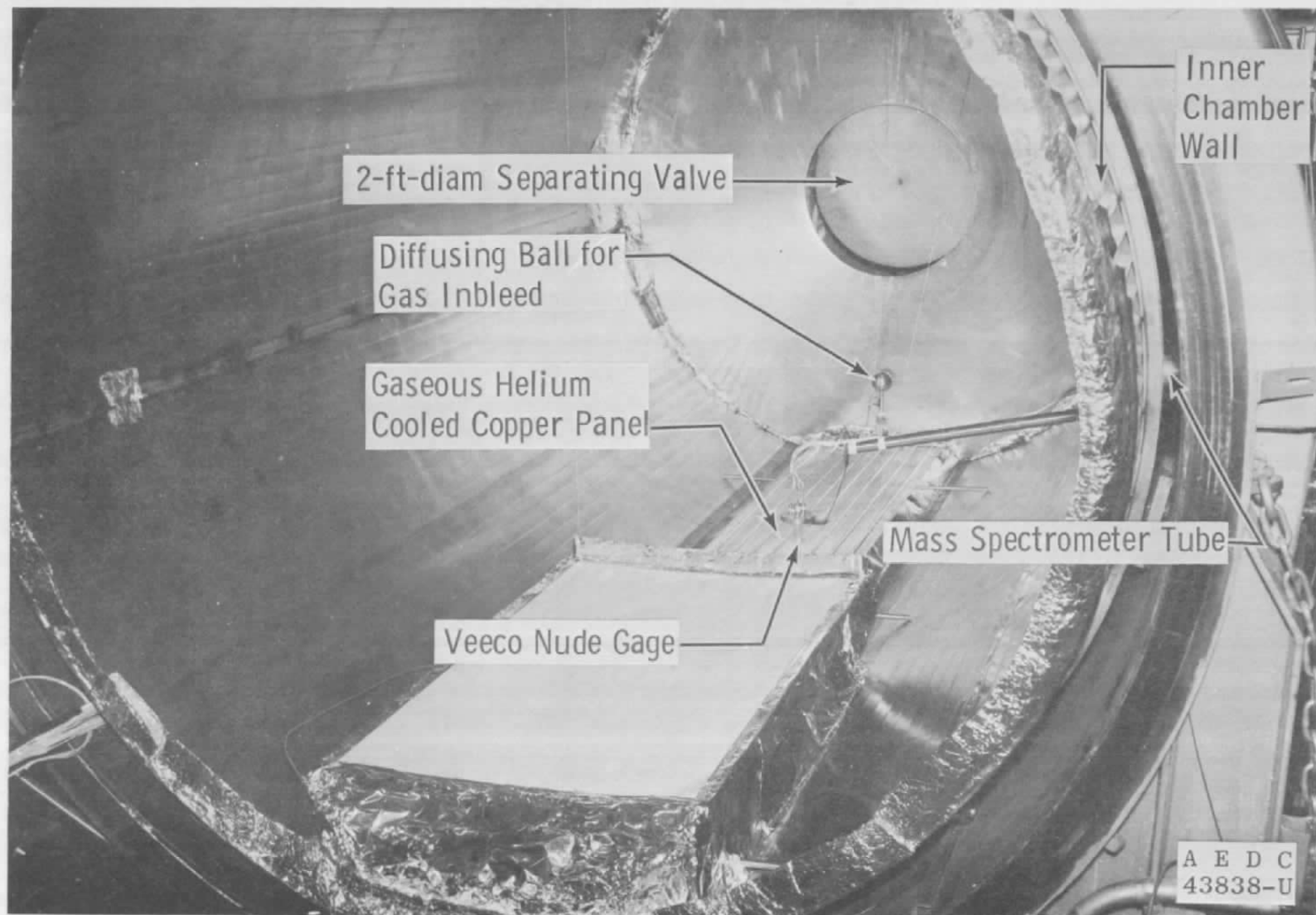
Fig. 5 Phase I Installation
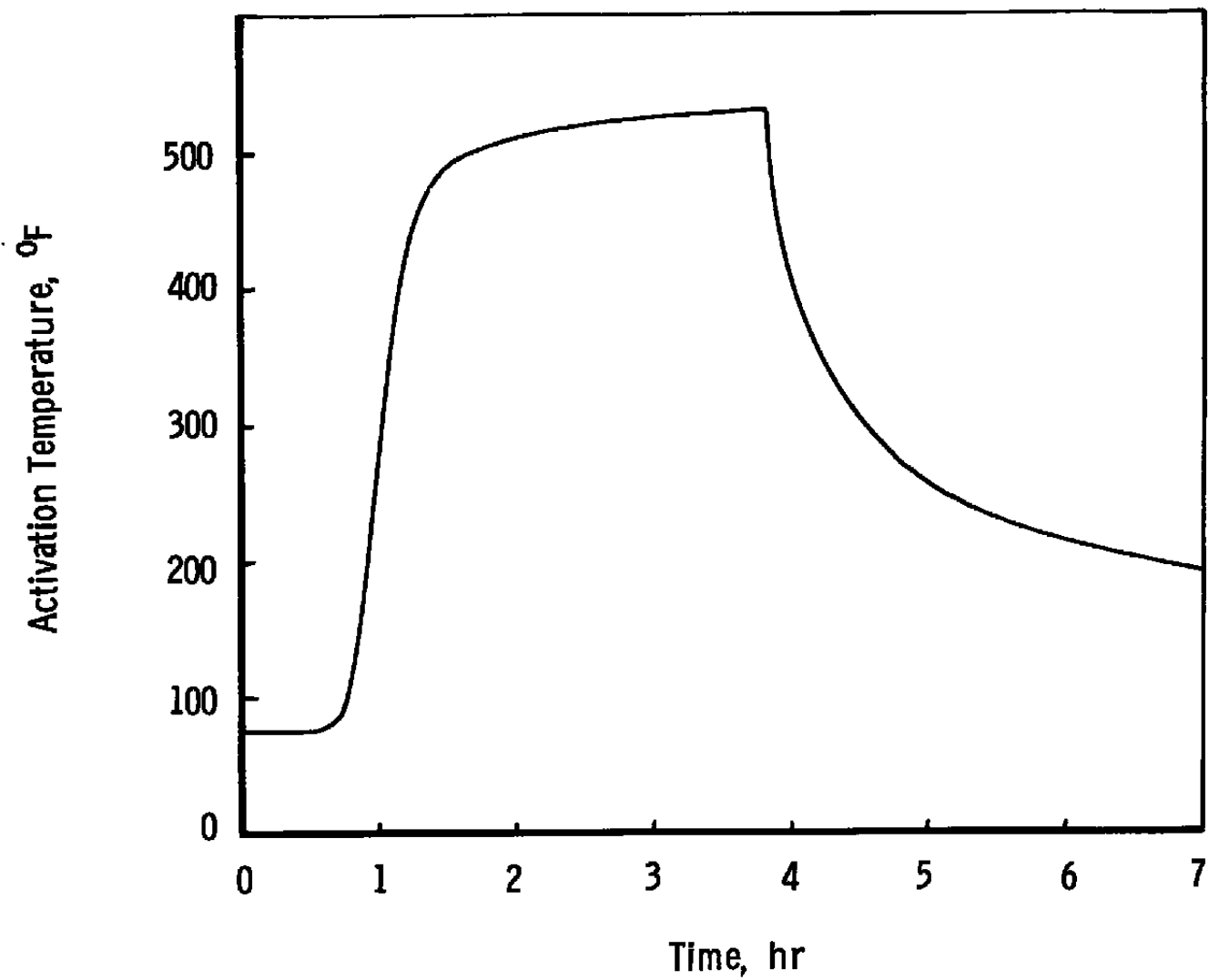
Fig. 6 Phase II Test Configuration, Runs 1-7

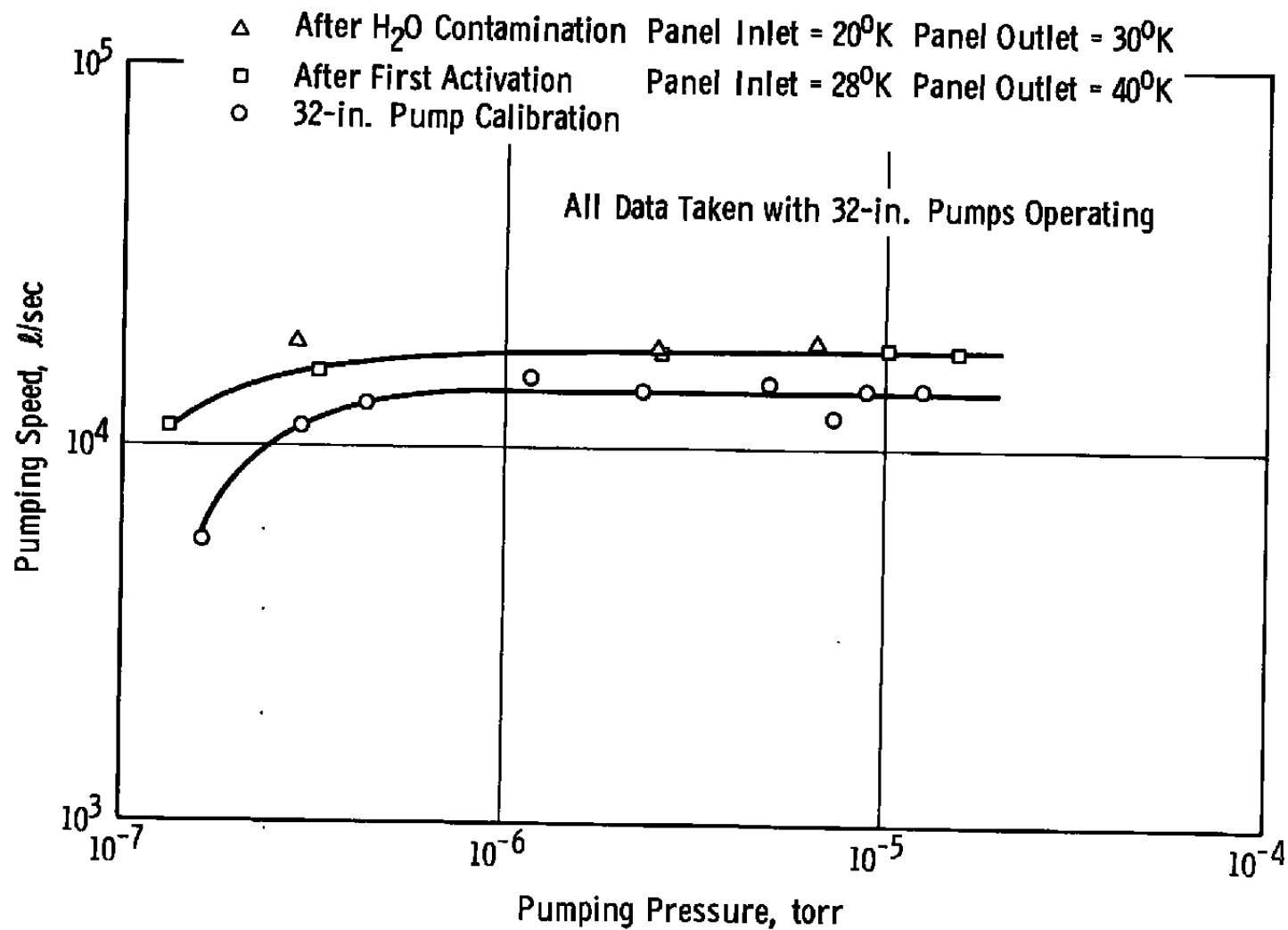Fig. 7 Typical Panel Activation Schedule

Fig. 8 77°K Hydrogen Pumping Speed versus Pumping Pressure for Molecular Sieve 5A and 32-in. Diffusion Pumps
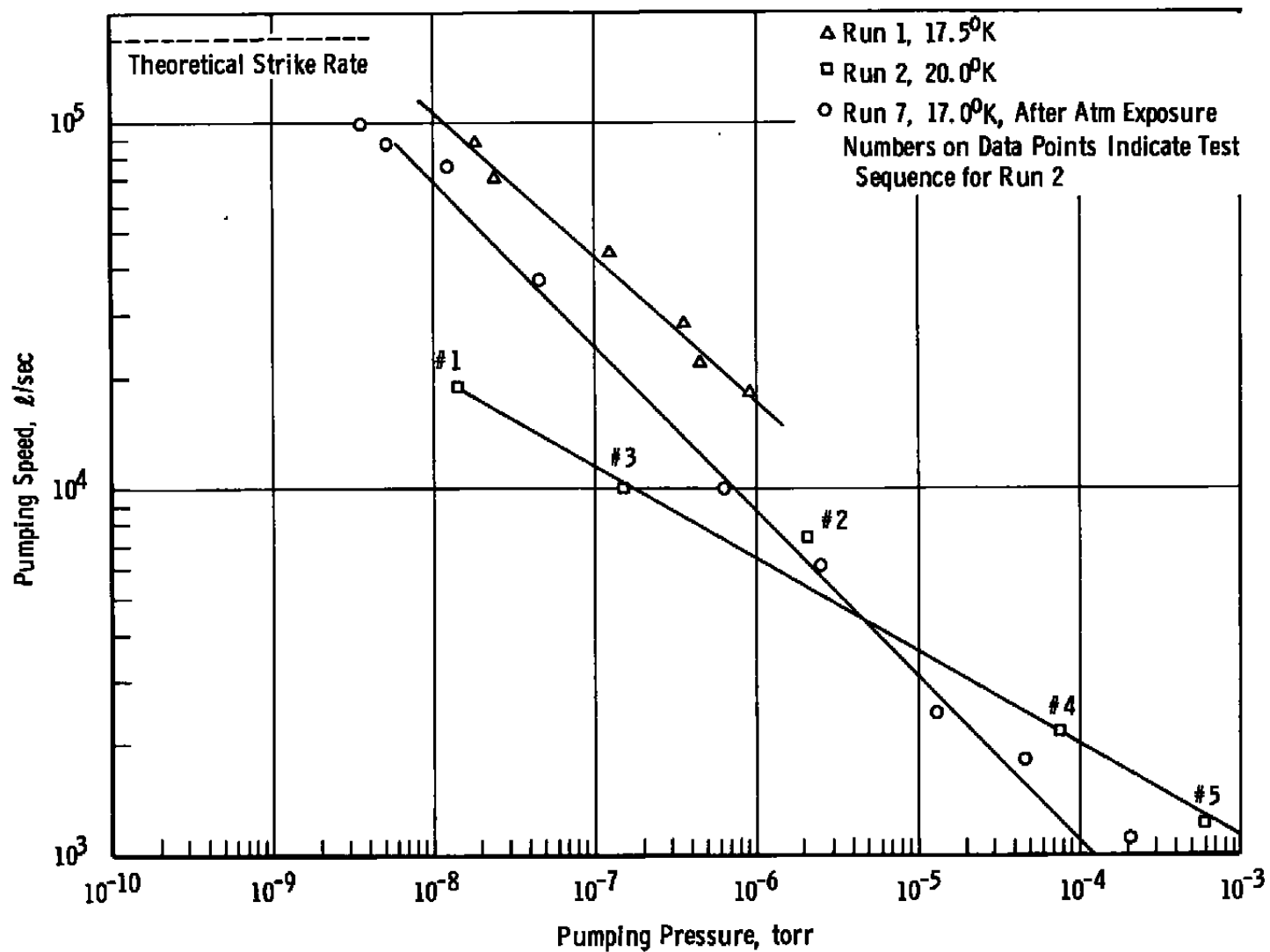
Fig. 9 Cryosorption Pumping of 77°K H₂ on Molecular Sieve 5A, Runs 1, 2, and 7
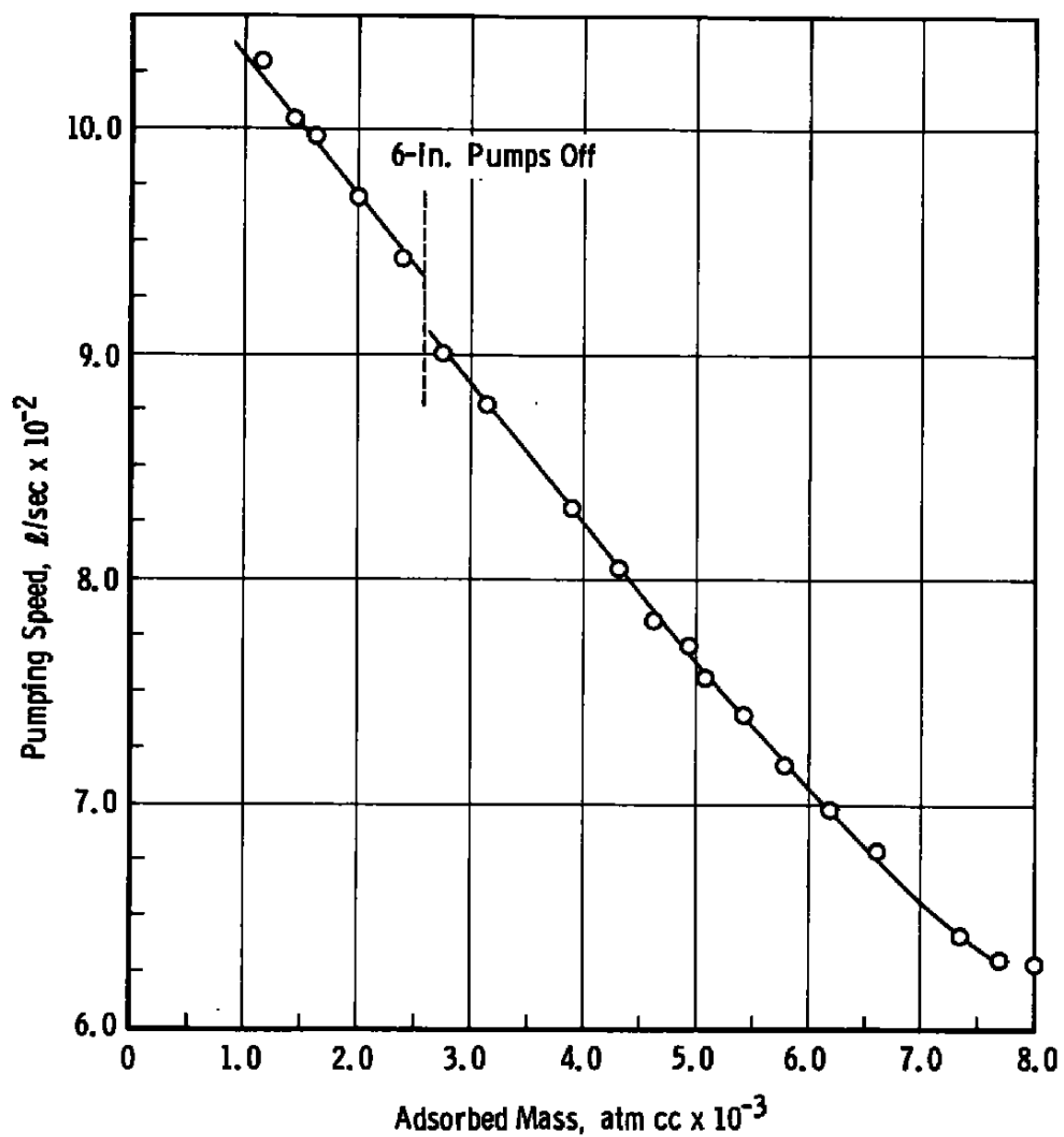
Fig. 10 Pumping Speed versus Mass Adsorbed, 77°K H$_2$ on 20°K Molecular Sieve 5A, Run 2
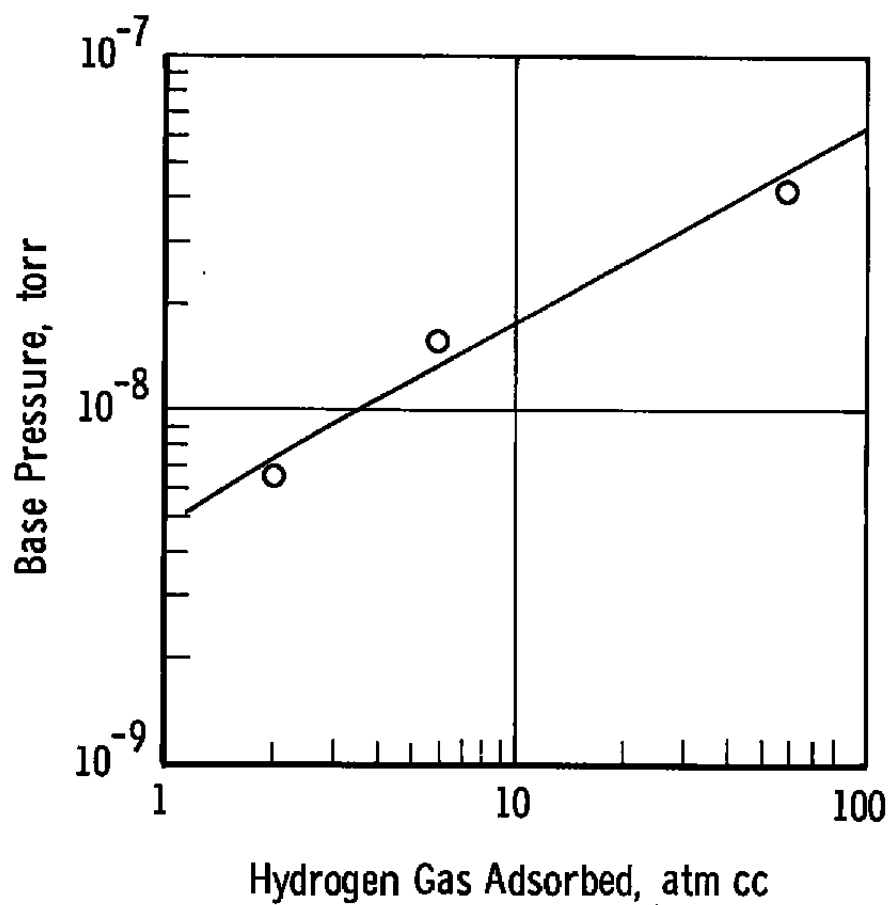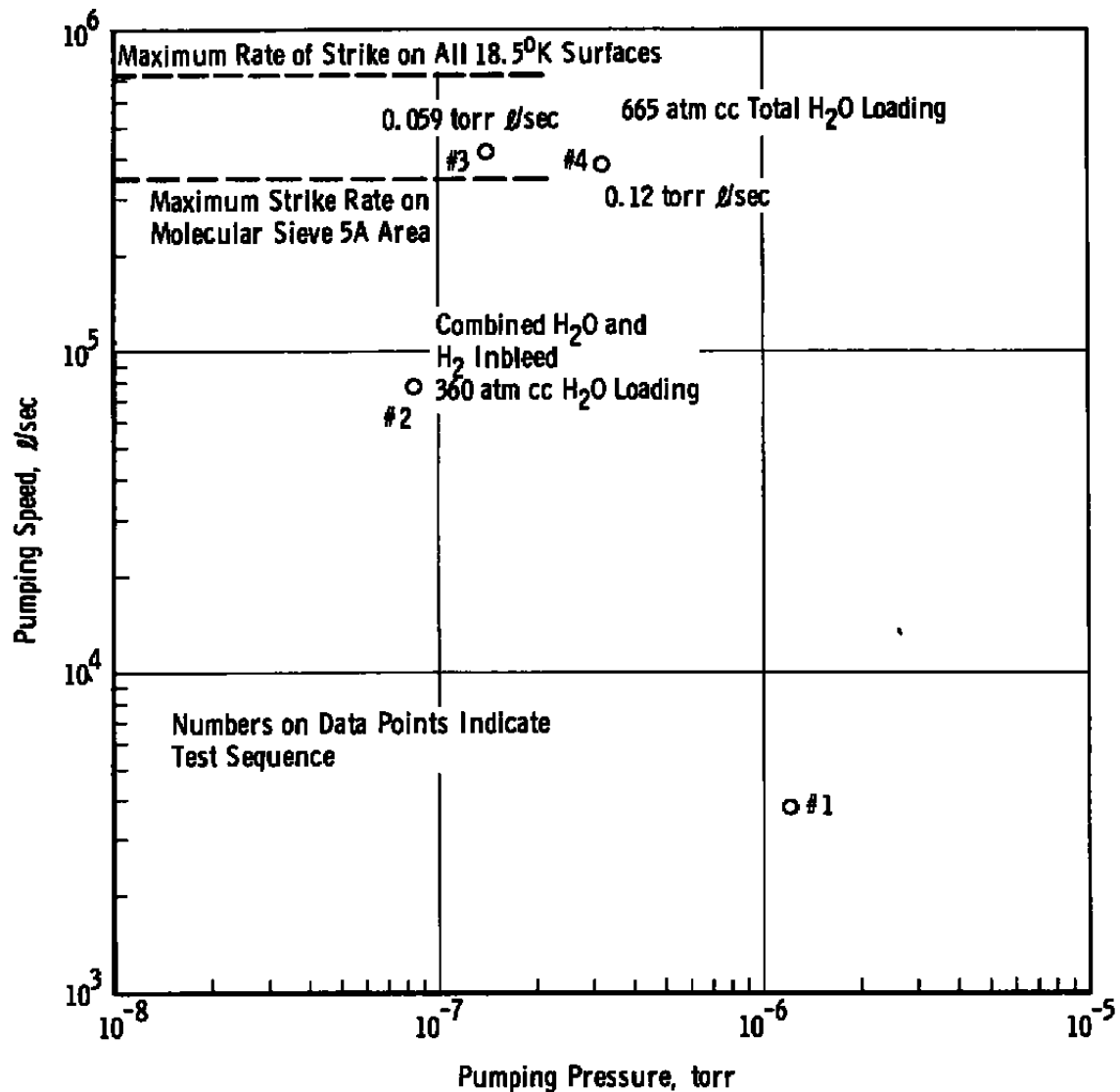
Fig. 11 17°K Isotherm for Molecular Sieve 5A, Run 7

Fig. 12 Cryosorption Pumping of 300°K H_2 on 18.5°K Molecular Sieve 5A, Water Contaminated, Run 3
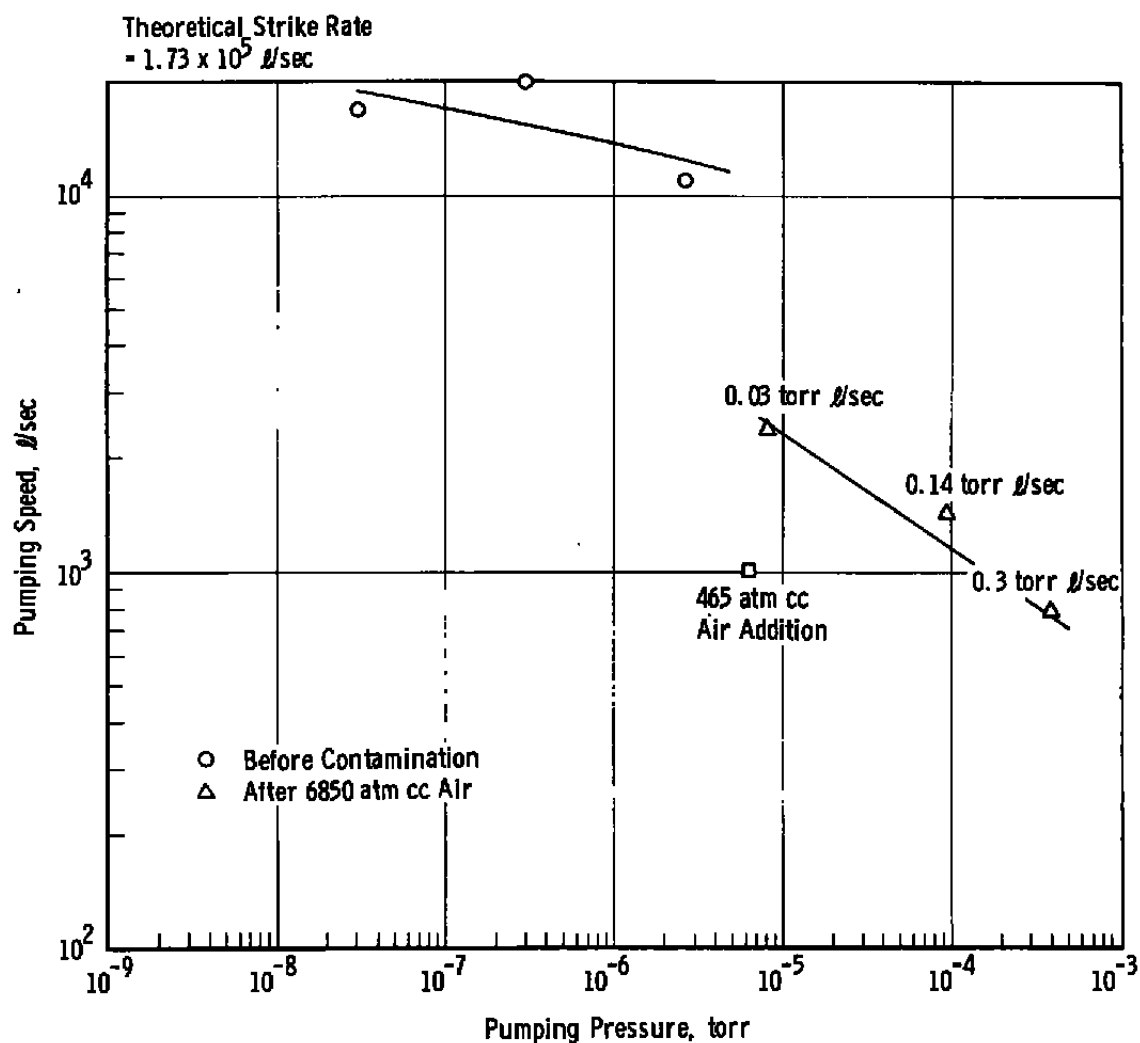
Fig. 13 77°K H_2 Pumping Speed on 16.4°K Molecular Sieve 5A, Air Contaminated, Run 4
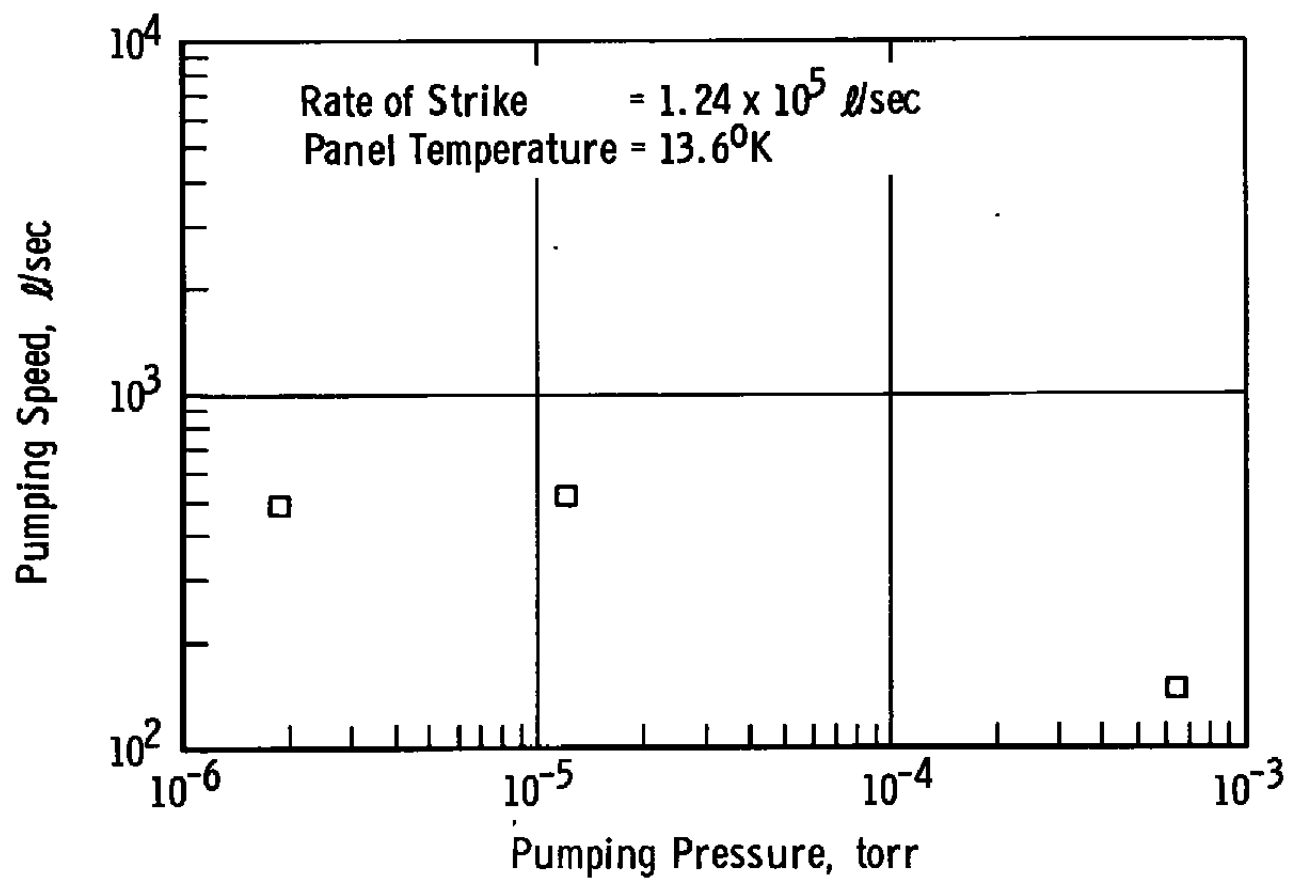
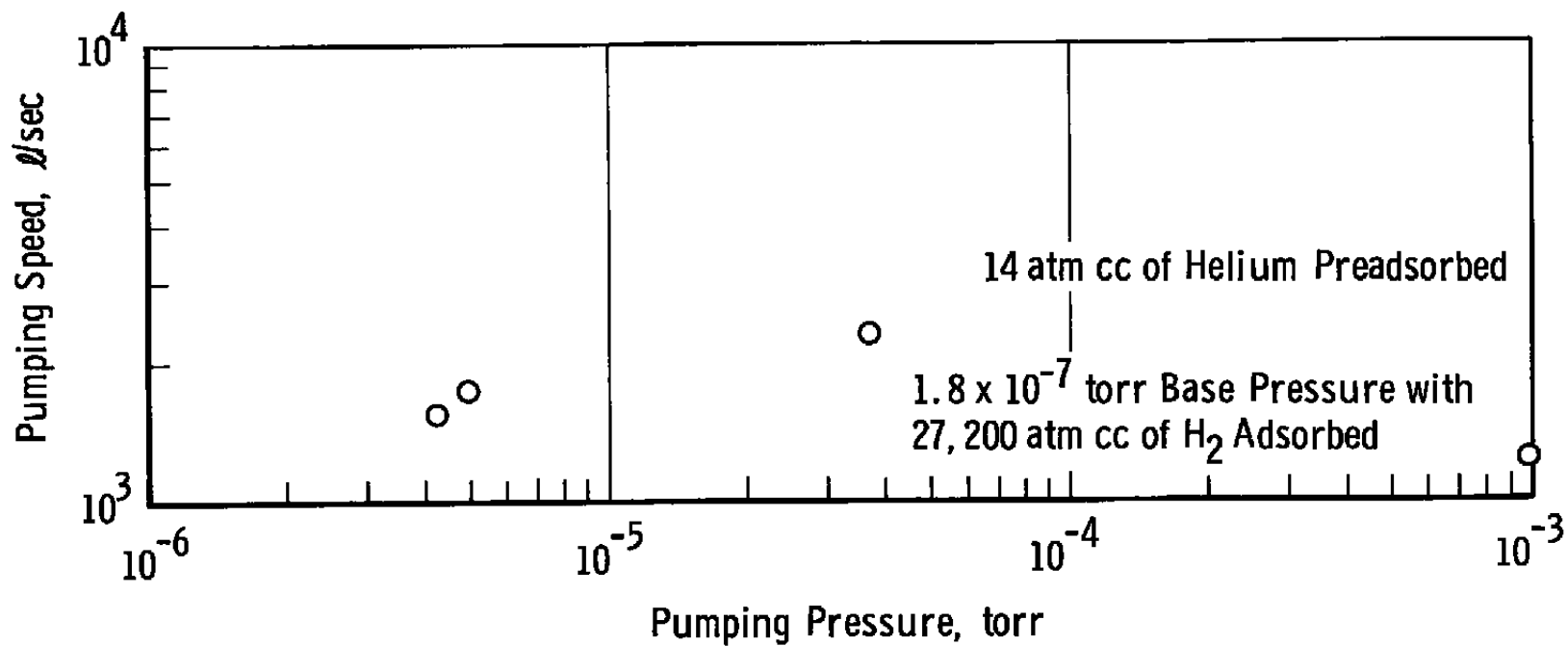Fig. 14 Cryosorption Pumping of 77°K He on 13.6°K Molecular Sieve 5A, Run 5

Fig. 15 Cryosorption Pumping of 77°K H₂ on 13.6°K Molecular Sieve 5A, Run 6

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13 ABSTRACT

Test results are presented for the cryosorption of hydrogen on a commercially manufactured molecular sieve 5A panel operated near 20°K. The effects of repeated activations, water and air contamination under vacuum, and exposure to atmospheric conditions were observed. Using suitable operating techniques, pumping speeds near theoretical maximums were obtained.

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14. KEY WORDS	LINK A		LINK B		LINK C	
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Molecular Sieve						
Helium						

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